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CELLULOSE FIBERS

HAVING LOW WATER RETENTION VALUE

AND LOW CAPILLARY DESORPTION PRESSURE

This application claims the benefit of U.S. Provisional Application No. 60/247,078, filed November 10, 2000, and U.S. Provisional Application No. 60/286,298, filed April 25, 2001, both of which are hereby incorporated by reference.

FIELD OF THE INVENTION

This invention relates to cellulose fibers having low water retention values and low median desorption pressures, as measured in a capillary absorption-desorption cycle, methods for preparing these fibers, and absorbent structures containing these fibers.

BACKGROUND OF THE INVENTION

Absorbent structures are important in a wide range of disposable absorbent articles including infant diapers, adult incontinence products, sanitary napkins and other feminine hygiene products and the like. These and other absorbent articles are generally provided with an absorbent core to receive and retain body liquids. In a conventional absorbent structure, the absorbent core is placed between a liquid pervious topsheet, whose function is to allow the passage of fluid to the core, and a liquid impervious backsheet whose function is to contain the fluid and to prevent it from passing through the absorbent article to the garment of the wearer of the absorbent article.

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An absorbent core for diapers, adult incontinence pads and feminine hygiene articles frequently includes fibrous batts or webs constructed of defiberized, loose, fluffed, hydrophilic, cellulosic fibers. Such fibrous batts form a matrix capable of absorbing and retaining some liquid. However, their ability to do so is limited. Thus, superabsorbent polymer (SAP) particles, granules, flakes or fibers (collectively particles), capable of absorbing many times their weight of liquid, are often included in the absorbent core to increase the absorbent capacity of the core, without having to substantially increase the bulkiness of the core. In an absorbent core containing matrix fibers and SAP particles, the fibers physically separate the SAP particles, provide structural integrity for the absorbent core, and provide avenues for the passage of fluid through the core.

Absorbent cores containing SAP particles have been successful, and in recent years, market demand has increased for thinner, more absorbent and more comfortable absorbent articles. As core absorbency has improved, the ability of the core to rapidly drain fluid from the absorbent article topsheet has become critical to maintaining a dry environment between the skin of the wearer of the absorbent article and the topsheet of the article.

The ability of the absorbent core to drain fluid from the layer immediately above it in the absorbent structure is controlled by gravity, by the number, size and spatial orientation of unoccupied volumes (voids or pores) in the absorbent core, and by the characteristics of the core components that impact fluid flow such as the wettability of the components by the acquired fluid, as indicated by contact angle, the surface tension of the acquired fluid and the viscosity of the acquired fluid. An acquisition layer, distribution layer, or acquisition and distribution layer can be included in the absorbent structure between the top sheet and absorbent core to facilitate draining of fluid into the absorbent core.

For optimum performance of an absorbent structure in terms of fluid capacity and core utilization, it is critical for fluids acquired by the absorbent core to move quickly from the moistened regions of the core to the dry regions of the core. The ability of the absorbent core to move fluids rapidly from a moistened region of the core to a dry region of the core may be described in terms of its permeability performance. The permeability of an absorbent core is defined as the ability of a liquid to flow through the absorbent core.

The ability of a first substrate, such as an absorbent core, to drain fluid primarily by capillary forces from a second substrate, such as an acquisition and distribution layer, is known as the partition property of the substrates.

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It is known to those skilled in the art that absorbent structures containing absorbent cores with good fluid partition properties also exhibit poor fluid permeability. Similarly, absorbent structures having good fluid permeability exhibit poor fluid partition properties. Consequently, it is important that fibers used in an acquisition layer have a higher

5   degree of stiffness or resiliency (measured as dry compressibility) under the weight of the diaper wearer than conventional fibers used in an absorbent core. This resiliency enables the interfiber spaces or voids in the acquisition layer to be maintained while the diaper is worn so that fluids can be quickly absorbed through the liquid permeable topsheet into the absorbent structure of the diaper.

10   It is also important for core fibers not to densify when wet, to the point that fluid flow into and through the absorbent core is restricted. In addition, core fibers must have sufficient physical integrity to maintain separation of wet SAP particles in an absorbent core so that as the particles swell, gel blocking is minimized or eliminated.

One method for increasing the stiffness and resiliency of fibers is by

15   crosslinking them. Cellulose fibers can be stiffened by intrafiber crosslinks, *i.e.*, crosslinks between two different portions of the same fiber, and to a lesser degree by interfiber crosslinks, *i.e.*, crosslinks between two different fibers.

Intrafiber crosslinking with certain aliphatic and alicyclic C_2-C_9 polycarboxylic acids is disclosed in U.S. Patent No. 5,190,563 issued to Herron *et al.* A "C_2-

20   C_9 polycarboxylic acid" as defined by Herron *et al.* is an organic acid containing two or more carboxyl groups and from 2 to 9 carbon atoms in the chain or ring to which the carboxyl groups are attached. Suitable C_2-C_9 polycarboxylic acids contain at least three carboxyl groups or two carboxyl groups with a carbon-carbon double bond present at the alpha, beta position relative to one or both carboxyl groups. When two carboxyl groups are separated by

25   a carbon-carbon double bond or are both connected to the same ring, the two carboxyl groups must be in the *cis* configuration. Examples of such polycarboxylic acids include citric acid, 1,2,3-propanetricarboxylic acid, 1,2,3,4-butanetetracarboxylic acid (BTCA), and oxydisuccinic acid. Herron *et al.* also found that cellulosic fibers crosslinked with aliphatic alkanes containing 4 carboxyl groups, namely, BTCA, had lower water retention values than

30   those containing 3 carboxyl groups, namely, citric acid and 1,2,3-propane tricarboxylic acid. Typically, fibers with lower water retention values are stiffer than those having higher water retention values.

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In contrast to cellulosic fibers having intrafiber crosslinks, cellulosic fibers having interfiber crosslinks, such as those found in most papers, are stiff when dry but do not necessarily maintain their stiffness when wet. Interfiber crosslinking of paper with citric acid and 1,2,3,4-butanetetracarboxylic acid and fabrics with maleic acid, citric acid, and 1,2,3,4-

5   butanetetracarboxylic acid is disclosed in D.F. Caulfield, *TAPPI J.*, 77(3): 205-212 (1994); D. Horie & C.J. Biermann, *TAPPI J.*, 77(8):135-140 (1994); Y.J. Zhou, P. Luner & P. Caluwe, *J. Appl. Polymer Sci.*, 58:1523-1534 (1995); and D. D. Gagliardi and F. B. Shippee, Am. Dyestuff Reptr., 52:300 (1963).

10   Zhou *et al.*, *supra*, studied the wet strength of paper crosslinked (interfiber) with certain polycarboxylic acids. Generally, interfiber crosslinking increases the wet strength of paper fibers. Zhou *et al.* found that the wet strength of the paper increased as the functionality of the polycarboxylic acid (*i.e.* the number of carboxyl groups in the polycarboxylic acid) increased. For example, 1,2,3,4-butanetetracarboxylic acid (BTCA) (4 carboxyl groups) was found to be more effective than tricarballylic acid (TCA) (3 carboxyl

15   groups), which in turn was found to be significantly more effective than succinic acid (2 carboxyl groups). Paper treated with succinic acid exhibited very little wet strength.

H. J. Campbell and T. Francis, *Textile Res. J.*, 35:260 (1965), crosslinked cotton cellulose with specific polycarboxylic acids. The reaction was catalyzed with trifluoroacetic anhydride (TFAA), necessitating the use of a non-aqueous solvent, in this case

20   benzene, to prevent hydrolysis of the TFAA. Campbell and Francis reported that succinic acid and glutaric acid showed only slight reactivity with cotton cellulose. Furthermore, they reported that esterification (or crosslinking) did not take place with oxalic acid. Malonic acid was found to react readily with cotton cellulose producing fabrics which were yellowed to an extent depending upon the degree of reaction.

25   Frequently, crosslinked cellulosic fibers are manufactured at a location remote from where they are incorporated into absorbent structures. Since the crosslinked fibers are bulky and have little fiber to fiber contact, they do not bond well to one another. Hence, sheets formed from crosslinked fibers fall apart easily. As a result, crosslinked cellulosic fibers are generally shipped in bales. This increases the cost of shipping the crosslinked

30   fibers and the cost of manufacturing the absorbent structure. It would, therefore, be desirable to prepare sheets of cellulosic fibers containing a crosslinking agent.

A "crosslinkable" cellulosic fibrous product formed into a web or sheet is disclosed in International Publication No. WO 00/65146. The crosslinkable product is

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formed by applying a crosslinking agent to a mat of cellulosic fibers and then drying the treated mat (without heating to a temperature sufficient to cure the crosslinking agent) such that substantially no crosslinking occurs and the product is substantially free from crosslinks.

U.S. Patent No. 6,059,924 disclose a process for enhancing the dry compression characteristics and the wicking property of fluff pulp. The process includes mildly refining a chemical pulp slurry prior to formation of a fluff pulp sheet.

There is a continuing need for improved cellulose fibers that have low water retention values and low median desorption pressures for incorporation into acquisition, distribution, and acquisition-distribution layers. There is also a need for core or matrix fibers that facilitate fluid flow into and through the absorbent core and maintain sufficient physical integrity to minimize or eliminate the gel blocking of swollen SAP particles. Finally, there is a need for methods of preparing sheets of crosslinkable cellulosic fibers.

SUMMARY OF THE INVENTION

This invention provides cellulose fibers having low median desorption pressures, as measured in a capillary absorption-desorption cycle, and low water retention values (WRV), which exhibit improved drainage and fluid flow properties. These fibers are particularly well suited for use in acquisition, distribution, and acquisition-distribution layers, and in absorbent core structures.

According to one embodiment, the fibers of the present invention are crosslinked and have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 15 cm or less. Preferably, the cellulose fibers also have a WRV of 45% or less. These fibers may be prepared by refining cellulose fibers to a freeness ranging from about 300 to about 700 ml Canadian Standard Freeness (CSF) and crosslinking the refined fibers. According to a preferred embodiment, the fibers are crosslinked with citric acid after refining.

Another embodiment of the invention is fibers crosslinked with at least one saturated dicarboxylic acid, aromatic dicarboxylic acid, cycloalkyl dicarboxylic acid, bifunctional monocarboxylic acid, or amine carboxylic acid. A crosslinking facilitator, such as oxalic acid, may be present during the crosslinking reaction to improve the efficacy of the crosslinking agent. According to one preferred embodiment, the cellulose fibers are refined prior to crosslinking in order to further stiffen them.

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Another embodiment is a method of preparing crosslinkable cellulose fibers comprising the steps of (a) crosslinking cellulose fibers with at least one crosslinking agent selected from saturated dicarboxylic acids, aromatic dicarboxylic acids, cycloalkyl dicarboxylic acid, bifunctional monocarboxylic acids, and amine carboxylic acids and (b) uncrosslinking the crosslinked cellulose fibers. Preferably, the crosslinking agent in this embodiment contains 4 carbon atoms or less. Two preferred crosslinking agents are oxalic acid and sodium chloroacetate. The crosslinkable fibers can be formed into sheets to ease their transport. Furthermore, the crosslinkable fibers can be re-crosslinked by curing the uncrosslinked cellulose fibers.

Yet another embodiment of the present invention is an acquisition, distribution, or acquisition and distribution layer comprising the cellulose fibers of the present invention.

Yet another embodiment is an absorbent core comprising cellulose fibers of the present invention. The absorbent core exhibits improved fluid flow properties into and through the core. According to one preferred embodiment, the absorbent core comprises SAP particles and reversible crosslinked fibers. The reversible crosslinked fibers separate the SAP particles and provide channels for fluid flow around the SAP particles from the wet to the dry areas of the absorbent core. Additionally, the reversible crosslinked fibers facilitate absorption of large volumes of urine (or other fluid) over a short period of time (e.g., a gush). Once urine or other fluid enters the absorbent core, the crosslinked fibers begin to uncrosslink. The uncrosslinked fibers hold and retain the urine or other fluid to a greater extent than fibers that are permanently crosslinked. As a result, the absorbent core has improved initial gush capacity compared to absorbent cores containing conventional fluff fibers and improved rewet performance compared to absorbent cores containing permanently crosslinked fibers.

Yet another embodiment is an absorbent structure comprising the acquisition, distribution, or acquisition and distribution layer of the present invention and/or the absorbent core of the present invention. Preferably, the absorbent structure contains a top (acquisition, distribution, or acquisition and distribution) layer and a bottom (storage) layer in fluid communication with the top layer. The absorbent structure exhibits superior partitioning from the acquisition and/or distribution layer to the storage layer compared to conventional absorbent structures.

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Yet another embodiment is an absorbent article containing the absorbent structure of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

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Definitions

The term "capillary absorption-desorption cycle" (also known as the capillary sorption cycle or CSC) refers to the process of determining the relationship between the pore volume of an absorbent structure and capillary pressure during absorption of a liquid into the

10 absorbent structure and subsequent drainage of the liquid from the absorbent structure. The capillary absorption-desorption cycle is indicative of the ability of an absorbent structure to attract, retain, and distribute fluid in the pores between the fibers of an absorbent structure. An absorbent structure may be subjected to this cycle by systematically lowering or raising the capillary pressure in narrow intervals, such as by the method described in the examples of

15 this application; "Capillary Sorption Equilibria in Fiber Masses", A. A. Burgeni and C. Kapur, *Textile Research Journal*, 37:356-366 (1967); and P.K. Chatterjee, Absorbency, Textile Science and Technology 7, Chapter II, pp. 63-65, Elsevier Science Publishers (1985), which are hereby incorporated by reference.

The "median desorption pressure" as determined in a capillary-desorption

20 cycle refers to the water swollen cellulose fibers ability to release water. For example, a sample of cellulose fibers that strongly retains water exhibits a much higher median desorption pressure than a sample of swollen cellulose fibers that readily releases water. The median desorption pressure as discussed herein is determined by the method described in the examples of this application; "Capillary Sorption Equilibria in Fiber Masses", A. A. Burgeni

25 and C. Kapur, *Textile Research Journal*, 37:356-366 (1967); and P.K. Chatterjee, Absorbency, Textile Science and Technology 7, Chapter II, pp. 63-65, Elsevier Science Publishers (1985), which are hereby incorporated by reference. This test method measures the ability of water swollen cellulose fibers to retain water against hydrostatic pressure.

The "water retention value" (WRV) of a cellulose fiber may be determined by

30 the methods described in TAPPI Useful Methods, UM 256, and P.K. Chatterjee, Absorbency, Textile Science and Technology Z, Chapter II, pp. 62-63, Elsevier Science Publishers (1985), which are both hereby incorporated by reference. The test measures the weight of water remaining in a sample of water saturated cellulose fibers after centrifugation and expresses

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that quantity as a weight percent based on the dry weight of the fibers. The WRV of a cellulose fiber is related to its drainage ability.

Any "cellulose fibers" known in the art, including cellulose fibers of any natural origin, such as those derived from wood pulp, may be used as starting materials in the methods of the present invention. Preferred cellulose fibers include, but are not limited to, digested fibers, such as kraft, prehydrolyzed kraft, soda, sulfite, chemi-thermal mechanical, and thermo-mechanical treated fibers, derived from softwood, hardwood or cotton linters. More preferred cellulose fibers include, but are not limited to, kraft digested fibers, including prehydrolyzed kraft digested fibers.

Generally, cellulose fibers having thicker walls are preferable since they are coarser and stiffer than similar fibers having thinner walls. The fiber walls of a fiber are defined by the lumen of a fiber (*i.e.* the hollow interior of the fiber) and the outer surface of the fiber. For example, since the fiber walls of southern softwoods are on average thicker than those of northern softwoods, fibers derived from southern softwoods are preferable. More preferably, the cellulose fibers are derived from softwoods, such as pines, firs, and spruces.

Other suitable cellulose fibers include those derived from Esparto grass, bagasse, kemp, flax and other lignaceous and cellulosic fiber sources. The cellulose fibers may be supplied in slurry, unsheeted or sheeted form.

The optimum fiber source utilized in conjunction with this invention will depend upon the particular end use contemplated. Generally, pulp fibers made by chemical pulping processes are preferred. Completely bleached, partially bleached and unbleached fibers are applicable. It may frequently be desirable to utilize bleached pulp for its superior brightness and consumer appeal. For products, such as paper towels, and absorbent pads for diapers, sanitary napkins, catamenials, and other similar absorbent paper products, it is especially preferred to use cellulose fibers derived from southern softwood pulp due to their premium absorbency characteristics.

More preferred cellulose fibers include, but are not limited to, bleached Kraft southern pine fibers sold under the trademark Foley Fluff™ which are available from Buckeye Technologies Inc. of Memphis, TN.

The cellulose fibers may have any fiber length. Typically, longer fibers produce crosslinked cellulose fibers having lower desorption pressures and water retention values than those produced from shorter fibers.

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<u>Refined and Crosslinked Fibers</u>

It has been surprisingly and unexpectedly discovered that when cellulose fibers are refined and crosslinked, the resulting fibers have low median desorption pressures as measured in a capillary absorption-desorption cycle and low water retention values (WRVs). Furthermore, these fibers exhibit improved fluid drainage in acquisition and/or distribution layers compared to similar unrefined fibers.

The cellulose fibers are crosslinked and have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 15 cm or less. Without being bound by any theory, the inventors believe this property is the result of intrafiber crosslinking within the cellulose fibers. More desirably, the cellulose fibers of this invention have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 14 cm or less; still more desirably, the fibers of this invention have a median desorption pressure, as determined in a capillary absorption-desorption cycle of 13 cm or less; and still more desirably, the fibers of the invention here a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 12 cm or less.

The refined and crosslinked cellulose fibers typically have a WRV of 45 percent or less; more desirably, 38 percent or less; and still more desirably 30 percent or less.

The cellulose fibers may be prepared by refining cellulose fibers to a freeness ranging from about 300 to about 700 ml CSF and crosslinking the refined fibers. According to one preferred embodiment, the starting cellulose fibers to be refined are wet lap. According to another preferred embodiment, the cellulose fibers are bleached and/or fluffed prior to being refined. The refined fibers may be crosslinked by any method known in the art, e.g., by reacting the fibers with a crosslinking agent.

Fibers having improved median desorption pressures and water retention values can be produced by refining the fibers first and then crosslinking the fibers with any of a wide variety of crosslinking agents. Suitable crosslinking agents include, but are not limited to, those described below as well as other polycarboxylic acids, such as aliphatic and alicyclic C_2-C_9 polycarboxylic acids. As used herein, the term "C_2-C_9 polycarboxylic acid" refers to an organic acid containing two or more carboxyl (COOH) groups and from 2 to 9 carbon atoms in the chain or ring to which the carboxyl groups are attached. The carboxyl groups are not included in determining the number of carbon atoms in the chain or ring. For example, 1,2,3-propanetricarboxylic acid would be considered to be a C_3 polycarboxylic acid

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containing three carboxyl groups. Similarly, 1,2,3,4-butanetetracarboxylic acid would be considered to be a C_4 polycarboxylic acid containing four carboxyl groups.

The C_2-C_9 polycarboxylic acids suitable for use as cellulose crosslinking agents in the present invention preferably include aliphatic and alicyclic acids either 5 olefinically saturated or unsaturated with at least three and preferably more carboxyl groups per molecule if a carbon-carbon double bond is present alpha, beta to one or both carboxyl groups. Additionally, in order to be reactive in esterifying cellulose hydroxyl groups, a given carboxyl group in an aliphatic or alicyclic polycarboxylic acid is preferably separated from a second carboxyl group by no less than two carbon atoms and no more than three 10 carbon atoms. Without being bound by theory, it appears that for a carboxyl group to be reactive, it must be able to form a cyclic 5 or 6-member anhydride ring with a neighboring carboxyl group in the polycarboxylic acid molecule. Where two carboxyl groups are separated by a carbon-carbon double bond or are both connected to the same ring, the two carboxyl groups must be in the *cis* configuration relative to each other if they are to interact 15 in this manner.

Novel Crosslinked Fibers

Another embodiment of the present invention is cellulose fibers crosslinked 20 with at least one saturated dicarboxylic acid, aromatic dicarboxylic acid, cycloalkyl dicarboxylic acid, bifunctional monocarboxylic acid, or amine carboxylic acid which exhibit low median desorption pressures as measured in a capillary absorption-desorption cycle and low water retention values. These crosslinked fibers exhibit improved fluid drainage in acquisition and/or distribution layers and improved permeability in absorbent cores.

25 Generally, these crosslinked cellulose fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 25 cm or less. Without being bound by any theory, it is believed that this property is the result of intrafiber crosslinking within the cellulose fibers.

More desirably, the crosslinked cellulose fibers have a median desorption 30 pressure, as determined in a capillary absorption-desorption cycle, of 20 cm or less; still more desirably, the fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 18 cm or less; still more desirably, the fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 15 cm or

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less; still more desirably, the fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 14 cm or less; still more desirably, the fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 13 cm or less; and still more desirably, the fibers have a median desorption pressure, as determined in a capillary absorption-desorption cycle, of 12 cm or less.

The crosslinked cellulose fibers typically have a WRV of 50 percent or less; more desirably, 45 percent or less; still more desirably, 38 percent or less; and still more desirably 30 percent or less.

The crosslinked cellulose fibers generally have a saturated capacity as measured by the procedure described in the examples of this application; Burgeni *et al.*, *supra*; and Chatterjee *et al.*, *supra*, of at least 10 grams of saline per gram of sample (g/g). According to a preferred embodiment, the crosslinked cellulose fibers have a saturated capacity of at least 11, 12, 13, 14, or 15 g/g.

The crosslinked cellulose fibers of the present invention are prepared by crosslinking cellulose fibers with one or more of the crosslinking agents of the present invention. The median desorption pressure and water retention value of the crosslinked fibers may be reduced by refining the fibers prior to crosslinking them. Furthermore, the crosslinking reaction may be performed in the presence of one or more of the crosslinking facilitators of the present invention to improve the efficacy of the crosslinking agents.

Refining

Refining may be performed by any method known in the art, including mechanical refining. Pulp refining involves application of work onto fibers, generally but not exclusively carried out in an aqueous slurry. For example, the fibers may be refined by cutting them, thereby reducing the average fiber length. Alternatively, the fibers may be refined by rubbing the fibers against each other and irregular surfaces under force or pressure. This causes the exterior fiber surfaces to increase in area, due to scoring and abrasion of the surfaces. In addition, the work put into the fibers during refining causes delamination of the interior fiber walls and surfaces. The result is weakened fiber walls that allow the fibers to absorb more water and swell to a greater extent than unrefined fibers. Never-dried refined fibers are also more flexible than similar unrefined never-dried fibers. Furthermore, when consolidated into a sheet, refined fibers, after drying, produce greater strength and stiffness in the sheet than is produced in a sheet of dried unrefined fibers.

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Methods of refining cellulose fibers, including, but not limited to, beating and fibrillation, are described in J. d'A. Clark, Pulp Technology and Treatment for Paper, 2nd Ed., Chapter 8, pp. 160-183, Chapter 12, pp. 277-305, Chapter 13, pp. 306-355, Chapter 14, pp. 356-407, Miller Freeman Pub., San Francisco (1985). A preferred method of refining cellulose fibers is fibrillation. The cellulose fibers may be refined with, for example, a disc refiner or a Valley beater available from Valley Mill Corporation of Lee, MA. Refining is typically performed at ambient temperature and pressure. For example, cellulose fibers may be refined by running an aqueous slurry of cellulose fibers through a Valley beater for 15 minute intervals until the desired freeness is obtained.

Generally, the fibers are wetted or moistened prior to being refined. According to one preferred embodiment, the cellulose fibers are bleached prior to being refined.

The cellulose fibers are broadly refined to a freeness of from about 300 to about 700 ml CSF and preferably to a freeness of from about 500 to about 700 ml CSF. According to a preferred embodiment, the cellulose fibers are refined to a freeness of from about 650 to about 700 CSF. The freeness of cellulose fibers as discussed herein is determined by TAPPI Method T-227.

Crosslinking

The refined or unrefined cellulose fibers are stiffened by intrafiber covalent crosslinking. Preferably, the cellulose fibers are wet or moist, prior to being reacted with the crosslinking agent and crosslinking facilitator. Desirably in some embodiments, the cellulose fibers are never-dried cellulose fibers.

The fibers are crosslinked by reacting them with a crosslinking agent and, optionally, a crosslinking facilitator of the present invention, such as those described below. Preferably, the fibers are crosslinked while in a highly twisted condition. Typically, the reaction step is performed under substantially unrestrained conditions, *i.e.*, individual fibers are free to move without interacting with neighboring fibers and are not under any substantial tension or pressure. The fibers may be reacted with the crosslinking agent and, optionally, a crosslinking facilitator by curing the fibers in the presence of the crosslinking agent and, optionally, the crosslinking facilitator.

Generally, the fibers are crosslinked by (i) mixing them with a crosslinking agent and, optionally, a crosslinking facilitator of the present invention and (ii) curing the

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fibers under conditions sufficient to cause intrafiber crosslinking. An effective amount of crosslinking agent and optionally crosslinking facilitator to cause formation of intrafiber crosslink bonds is typically mixed with the cellulose fibers. Preferably, an effective amount of crosslinking facilitator to increase the number or rate of intrafiber crosslink bonds formed

5   by the reaction of the fibers with the crosslinking agent is mixed with the cellulose fibers. Generally, from about 0.5 to about 40 mole percent and preferably from about 1 to about 30 mole percent of crosslinking agent and crosslinking facilitator, calculated on a cellulose anhydroglucose molar basis, is mixed with the fibers. When the crosslinking agent is a dicarboxylic crosslinking agent, generally from about 5 to about 21 mole percent of

10 crosslinking agent, calculated on a cellulose anhydroglucose molar basis, is mixed with the fibers. Generally, from about 1.8 to about 9 mole percent of crosslinking facilitator, calculated on a cellulose anhydroglucose molar basis, is mixed with the fibers. The mixture containing the fibers and crosslinking agent preferably contains from about 5 to about 10% by weight of crosslinking agent based upon the dry weight of the fibers.

15   After the crosslinking agent is mixed with the fibers, the fibers are preferably separated and individualized, by, for example, fluffing the fibers or disintegrating and fluffing the fibers. By separating the fibers, intrafiber crosslinking is maximized while interfiber crosslinking is minimized. Preferably, the fibers are crosslinked by formation of intrafiber covalent bonds.

20   Cellulose fibers supplied as wet lap, dry lap or other sheeted form may be separated by mechanically disintegrating them to unsheeted form. In the case of dry lap, it is advantageous to moisten the fibers, for example to 40% moisture (60% solids content, based on the total weight of fiber and water), prior to mechanical disintegration in order to plasticize the fibers and minimize damage to the fibers.

25   When the crosslinking agent is applied to the fibers in an aqueous solution, the fibers are dried prior to being cured. The fibers are preferably dried to remove all the water in the fibers and cured to establish intrafiber crosslinking. Drying may be performed by any method known in the art. Typically, drying is performed by heating the fibers at a temperature of from about 50 to about 225° C. Preferably, drying is performed at from about

30 105 to about 175° C. The fibers are typically dried to constant weight. The temperature of the fibers during the drying process generally does not exceed 100° C, the boiling point of water, irrespective of the temperature at which the fibers are dried, until all of the water has been evaporated from the fibers. As discussed in T. Lindstrom, Paper Structure and

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Properties, International Fiber and Technology Series 8, Chapter 5, pp 104-105, Marcel Dekker Inc., New York (1986), drying of cellulose fibers typically leads to an irreversible reduction in the swelling ability of the fibers on rewetting. This phenomenon is commonly referred to as hornification. Without being bound to any theory, it is believed that the microfibrils in the fiber walls bond together during the drying process thereby reducing the size of pores in the fiber walls. This results in stiffened fibers, compared to the fibers before drying. The subsequent curing stage facilitates formation of the intrafiber covalent bonds that lock in the dried fiber stiffness and geometry.

Curing is generally performed at a temperature sufficient to cause intrafiber covalent bonds to form. Curing is broadly performed at a temperature of from about 105 to about 225° C. Preferably, the cellulose fibers are cured at a temperature of from about 150 to about 190° C. More preferably, they are cured at a temperature of from about 160 to about 175° C. Curing may be performed for 15, 30, 45, or 60 minutes or longer.

According to one preferred embodiment, the fibers are crosslinked by (i) contacting an aqueous solution of the crosslinking agent and, optionally, crosslinking facilitator with an aqueous mixture containing the cellulose fibers, (ii) removing water from the aqueous mixture, (iii) mechanically separating the fibers into substantially individual form, (iv) drying the fibers, and (v) reacting the fibers with the crosslinking agent to cause crosslinking in the fibers. Generally, step (ii) involves removing the majority of water from the aqueous mixture. Preferably, enough water is removed from the aqueous mixture to obtain a mixture having from about 40 to about 80% by weight of solids, based upon 100% total weight of fibers and water. According to a more preferred embodiment, step (ii) involves removing water from the aqueous mixture to obtain a mixture having about 60% by weight of solids, based upon 100% total weight of fibers and water. The water removal, separating, and drying steps cause the fibers to become highly twisted. The twisted condition generally is at least partially, but less than completely, permanently set by the crosslinking reaction.

For example, the fibers may be crosslinked by the method described in U.S. Patent No. 5,190,563, which is hereby incorporated by reference, substituting the crosslinking agents and crosslinking facilitators of the present invention for the crosslinking agent in U.S. Patent No. 5,190,563. In U.S. Patent No. 5,190,563, cellulosic fibers are contacted with a solution containing a C_2-C_9 polycarboxylic acid crosslinking agent. The fibers are then mechanically separated into substantially individual form, dried, and reacted with the

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crosslinking agent while remaining in substantially individual form so that intrafiber crosslink bonds form. The individualized cellulosic fibers are contacted with an amount of crosslinking agent effective to cause the fibers to form intrafiber crosslink bonds. Preferably, from about 0.5 mole percent to about 6.0 mole percent crosslinking agent, calculated on a cellulose anhydroglucose molar basis, is contacted with the fibers.

When the crosslinking agent contains an amino or amine group to be reacted, the crosslinking agent is preferably activated prior to or simultaneously with the crosslinking reaction. The term "activated" as used herein refers to modifying the crosslinking agent so that the nitrogen atom of the amino or amine group is in a more reactive condition, *i.e.*, more prone to reaction. The crosslinking agent may be activated by any method known in the art. For example, the amine or amino containing crosslinking agent can be reacted with nitrous acid to activate the nitrogen atom of the amine or amino group.

The fibers may be crosslinked in the presence a reducing agent (antioxidant) to prevent yellowing of the fibers during the crosslinking reaction. Suitable reducing agents include, but are not limited to, hypophosphites, such as sodium hypophosphite; sodium bisulfite; sodium phosphite; and any combination of any of the foregoing. A preferred reducing agent is sodium hypophosphite.

The fibers may be bleached during or after the crosslinking reaction to improve their appearance. For example, the fibers may be bleached by reacting them with a bleaching agent. Any bleaching agent known in the art may be used. Suitable bleaching agents include, but are not limited to, hydrogen peroxide.

For example, the bleaching agent may be included in an aqueous solution containing the crosslinking agent that is applied to the fibers. Preferably, the aqueous solution contains a sufficient amount of bleaching agent so that the mixture obtained from adding the aqueous solution to the fibers contains from about 2.5 to about 5% by weight of bleaching agent, based on the dry weight of the fibers.

Saturated Dicarboxylic Acid Crosslinking Agents

The term "saturated dicarboxylic acid" refers to dicarboxylic acids that do not contain any carbon-carbon double or triple bonds. The saturated dicarboxylic acids may contain linear or branched aliphatic chains, *i.e.*, they are acyclic. Preferred saturated dicarboxylic acids include, but are not limited to, C_2-C_8 saturated dicarboxylic acids. The term "C_2-C_8 saturated dicarboxylic acid" refers to a dicarboxylic acid in which the total

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number of carbon atoms (including those in the carboxyl groups) ranges from 2 to 8. Non-limiting examples of C_2-C_8 saturated dicarboxylic acids are oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, and suberic acid. Special mention is made of C_2-C_6 saturated dicarboxylic acids and C_2-C_4 saturated dicarboxylic acids.

5 According to one preferred embodiment, C_3 and higher saturated dicarboxylic acids, such as C_3-C_8 saturated dicarboxylic acids, are applied to the cellulose fibers in conjunction with a crosslinking facilitator, such as oxalic acid.

Another class of saturated dicarboxylic acids is saturated hydroxy dicarboxylic acids. The term "saturated hydroxy dicarboxylic acid" refers to saturated dicarboxylic acids
10 that contain at least one hydroxy substituent. Suitable saturated hydroxy dicarboxylic acids include, but are not limited to, C_2-C_8 hydroxy saturated dicarboxylic acids (*i.e.* those containing from 2 to 8 carbon atoms). Special mention is made of C_2-C_8 polyhydroxy saturated dicarboxylic acids. Non-limiting examples of C_2-C_8 hydroxy saturated dicarboxylic acids are tartaric acid, malic acid, saccharic acid, and mucic acid.
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Aromatic Dicarboxylic Acid Crosslinking Agents

The term "aromatic dicarboxylic acid" refers to aromatic compounds having the formula HOOC-R-COOH wherein R is a substituted or unsubstituted phenyl group. The term "substituted" as used herein includes, but is not limited to, at least one of the following
20 substituents: hydroxy, C_1-C_4 alkoxy, C_1-C_4 alkyl, amino, halogen, and nitro.

A preferred aromatic dicarboxylic acid has the formula

25
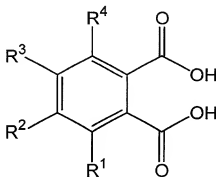
30 where R^1, R^2, R^3, and R^4 independently are hydrogen, hydroxy, C_1-C_4 alkoxy, C_1-C_4 alkyl, amino, halogen, or nitro. A preferred aromatic dicarboxylic acid is phthalic acid.

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Cycloalkyl Dicarboxylic Acid Crosslinking Agents

The term "cycloalkyl dicarboxylic acid" refers to cycloalkyl dicarboxylic acids that do not contain carbon-carbon double bonds in the α or β positions relative to the carboxyl groups. According to one embodiment, the cycloalkyl dicarboxylic acid has the formula
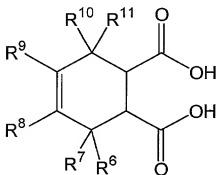
wherein

R^6, R^7, R^{10}, and R^{11} are independently hydrogen, hydroxy, halogen, C_1-C_4 alkoxy, C_1-C_4 alkyl, amino, or nitro; and

R^8 and R^9 are independently hydrogen, halogen, C_1-C_4 alkoxy, or C_1-C_4 alkyl.

A preferred cycloalkyl dicarboxylic acid is 1,2,5,6-tetrahydrophthalic acid.

Bifunctional Monocarboxylic Acid Crosslinking Agents

A "bifunctional monocarboxylic acid" refers to an organic acid having (a) only one carboxyl group and (b) a functional group, which is not a carboxyl group, capable of reacting with the carboxyl, carboxylic acid, amino, or hydroxyl groups of a polymer. Preferably, the bifunctional monocarboxylic acid includes only two functional groups, *i.e.*, a carboxyl group and a second functional group.

Suitable bifunctional monocarboxylic acids include, but are not limited to, amino acids, salts of haloacetates, hydroxy monocarboxylic acids, and acid derivatives of hydroxy monocarboxylic acids, such as acid esters of hydroxy monocarboxylic acids.

A preferred salt of a haloacetate is sodium chloroacetate. Without being bound by any theory, it is believed that when an aqueous mixture of sodium chloroacetate and cellulose fibers is dried and cured, an ether is formed by reaction of a cellulose hydroxyl group with the chlorine containing carbon of the sodium chloroacetate molecule. This etherification reaction releases a molecule of hydrochloric acid, which is immediately neutralized by the sodium salt of the newly formed cellulose based acid. At elevated

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temperature, this acid is available for esterification to proximate hydroxyl groups in the fibers, with concomitant release of water. After the ether and ester formation, sodium chloride remains as a byproduct.

Suitable hydroxy monocarboxylic acids and acid derivatives thereof include, but are not limited to, glycolic acid, methane sulfonic acid ester of glycolic acid, and para-toluene sulfonic acid ester of glycolic acid.

Amine Carboxylic Acid Crosslinking Agents

Suitable amine carboxylic acids include, but are not limited to, primary, secondary, and tertiary amines and aromatic amines. Preferred primary amines include, but are not limited to, amino acids. Special mention is made of amino acids having the formula

$$H_2N\text{-}CH_2\text{-}R^{12}\text{-}C(O)OH$$
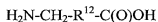
where R^{12} is a bond, $C_1\text{-}C_{12}$ alkyl, or a $C_1\text{-}C_{12}$ alkyl substituted with one or more of carboxyl, hydroxy, $C_1\text{-}C_4$ alkoxy, $C_1\text{-}C_4$ alkyl, amino, and nitro.

Preferred amino acids include, but are not limited to, those having the formula
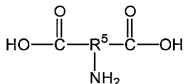
where R^5 is a linear or branched $C_1\text{-}C_8$ alkyl. According to a preferred embodiment, R^5 is a $C_2\text{-}C_4$ alkyl. Non-limiting examples of suitable amino acids include aspartic acid and glutamic acid.

Other suitable amine carboxylic acid crosslinking agents include, but are not limited to, ethylenedinitrilotetraacetic acid (EDTA).

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Crosslinking Facilitators

The crosslinking facilitators of the present invention increase the efficacy of the crosslinking agents. A preferred crosslinking facilitator is oxalic acid. Without being bound by any theory, it is believed that oxalic acid (pK_a = 1.23) may serve as an acid catalyst for esterification of the crosslinking agent. Alternatively, oxalic acid may form a mixed anhydride with the crosslinking agent which then facilitates esterification of the cellulose fibers.

Crosslinking Reversibility

Fibers crosslinked with the short crosslinking agents of the present invention, *i.e.*, those containing 4 carbon atoms or less (*e.g.* 3 carbon atoms or less), such as oxalic acid and sodium chloroacetate, can be uncrosslinked and crosslinked thereafter. The crosslinking of such fibers is generally substantially reversible, *i.e.*, at least about 50% by weight of the crosslinked fibers can be uncrosslinked. According to one embodiment, at least about 60, 70, 80, 90, or 95% by weight of the crosslinked fibers can be uncrosslinked.

The crosslinked fibers can be uncrosslinked by soaking them in water for a time sufficient to uncrosslink them. Typically, the crosslinked fibers are soaked for from about 0.5 to about 4 hours. According to a preferred embodiment, the fibers are soaked for about 2 hours. The crosslinked fibers can also be uncrosslinked by subjecting them to a capillary absorption-desorption cycle as described in the examples of this application; Burgeni *et al.*, *supra*; and Chatterjee *et al.*, *supra*.

The fibers can be re-crosslinked by drying or drying and curing them. This phenomenon was not observed with the covalently crosslinked fibers disclosed in U.S. Patent Nos. 5,137,537; 5,183,707 and 5,190,563.

Without being bound by any theory, it is believed that as the crosslinked fibers absorb water and swell, the crosslinks are strained as the cellulose polymer chains move apart to accommodate the absorbed water. When the crosslink molecules are very short in length, as with fibers treated with oxalic acid or sodium chloroacetate (two carbon atoms separate the hydroxyl groups on the neighboring cellulose polymer chains), the strain of the fibers swelling is sufficient to facilitate hydrolysis and cleavage of one of the two covalent bonds that crosslink the fibers. In contrast, a citric acid crosslinked fiber as disclosed in U.S. Patent Nos. 5,137,537; 5,183,707 and 5,190,563 has a much longer molecule bridging the space between cellulose polymer chains (four or five carbon atoms separate the hydroxyl groups on

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the neighboring cellulose chains). Consequently the strain on the longer crosslinking molecule as the fibers absorb water and swell, is not sufficient to facilitate cleavage of one of the crosslinking covalent bonds.

Since the fibers of the present invention can be uncrosslinked, they can be dried and transported or stored in sheeted form instead of in bulk or baled form. This reduces shipping and storage costs. The fibers can be re-crosslinked at the destination or whenever desired by, for example, separating and curing them. Once the fibers are re-crosslinked, they can, for example, be incorporated into an absorbent structure.

The terms "reversible crosslinked fibers" and "reversible crosslinked cellulose fibers" as used herein refer to crosslinked fibers or crosslinked cellulose fibers in which at least about 50, 60, 70, 80, 90, or 95% by weight of the crosslinked fibers become uncrosslinked after being soaked in water for up to 4 hours and in which at least 50, 60, 70, 80, 90, or 95% of the uncrosslinked fibers can re-crosslinked by drying the fibers at a temperature of 105° C or higher.

The reversible crosslinked fibers of the present invention are particularly suitable for use in an absorbent core containing superabsorbent polymer (SAP) particles. The crosslinked fibers separate the SAP particles and provide channels for fluid flow around the SAP particles from the wet to the dry areas of the absorbent core. Additionally, the reversible crosslinked fibers facilitate absorption of large volumes of urine (or other fluid) over a short period of time (e.g., a gush). Once urine or other fluid enters the absorbent core, the crosslinked fibers begin to uncrosslink. According to one embodiment, after 0.5 to 4.0 hours of exposure to the urine or other fluid, the majority of wet fibers are uncrosslinked. The uncrosslinked fibers hold and retain the urine or other fluid to a greater extent than fibers that are permanently crosslinked. As a result, the absorbent core has improved initial gush capacity compared to absorbent cores containing conventional fluff fibers and improved rewet performance compared to absorbent cores containing permanently crosslinked fibers.

Absorbent Structures

The cellulose fibers of the present invention may be incorporated into any disposable or non-disposable absorbent structure intended to absorb and contain body exudates, and which are generally placed or retained in proximity with the body of the wearer. Such absorbent structures are commonly employed in disposable and non-disposable absorbent articles. Examples of disposable absorbent articles include, but are not limited to,

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infant diapers, adult incontinence products, training pants, sanitary napkins and other feminine hygiene products. Examples of absorbent structures in which the cellulose fibers of the present invention may be incorporated include, but are not limited to, those described in International Publication Nos. WO 98/47456, WO 99/63906, WO 99/63922, WO 99/63923, WO 99/63925, WO 00/20095, WO 00/38607, WO 00/41882, WO 00/71790, and WO 00/74620 and U.S. Patent No. 5,695,486, all of which are hereby incorporated by reference.

Acquisition and Distribution Layers

The cellulose fibers of the present invention may be incorporated into an acquisition, distribution, or acquisition-distribution layer. Such layers are commonly employed in absorbent structures contained in disposable absorbent articles. The acquisition and/or distribution layer may be prepared and incorporated into an absorbent structure by any method known in the art. According to one embodiment, the absorbent structure comprises a top layer, which includes the acquisition and/or distribution layer of the present invention, and a bottom storage layer (also known as an absorbent core). The acquisition and distribution layer may be a single layer or two separate layers, *i.e.*, a top acquisition layer and a lower distribution layer. The lower distribution layer rapidly drains fluid from the acquisition layer and distributes the fluid into the storage layer.

The acquisition layer of the present invention typically includes from about 90 to about 100% by weight of the cellulose fibers of the present invention, based upon 100% total weight of acquisition layer. The density of the acquisition layer broadly ranges from about 0.04 to about 0.07 g/cm^3.

Absorbent Core

The cellulose fibers of the present invention may be incorporated into an absorbent core (also known as a storage layer). The absorbent core may include any material known in the art that absorbs liquid. Suitable materials include, but are not limited to, fibrous batts or webs constructed of defiberized, loose, fluffed, and/or hydrophilic cellulosic fibers or the fibers of the present invention; superabsorbent polymer (SAP) particles, granules, flakes or fibers (collectively particles); and any combination of the foregoing. Generally, SAP particles are capable of absorbing many times their weight of liquid and significantly increase the absorbent capacity of the absorbent core without substantially increasing the bulkiness of the layer.

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The term superabsorbent polymer particle or SAP particle is intended to include any particulate form of superabsorbent polymer, including irregular granules, spherical particles (beads), powder, flakes, staple fibers and other elongated particles. SAP refers to a normally water-soluble polymer which has been cross-linked to render it substantially water insoluble, but generally capable of absorbing at least ten, and preferably at least fifteen, times its weight of a physiological saline solution. The SAP particles may be of any size or shape. Numerous examples of superabsorbers and their methods of preparation may be found, for example, in U.S. Patent Nos. 4,102,340; 4,467,012; 4,950,264; 5,147,343; 5,328,935; 5,338,766; 5,372,766; 5,849,816; 5,859,077; and Re. 32,649. Examples of suitable SAP particles include, but are not limited to, starch graft copolymers, such as hydrolyzed starch-acrylate graft co-polymer; cross-linked carboxymethylcellulose and derivatives thereof; and modified hydrophilic polyacrylates, such as saponified acrylic acid ester-vinyl co-polymer, neutralized crosslinked polyacrylic acid, and cross-linked polyacrylate salts.

Preferably, the SAP particles form hydrogels upon absorbing fluid. More preferably, the SAP particles have high gel volumes or high gel strength as measured by the shear modulus of the hydrogel. Such SAP particles typically contain relatively low levels of polymeric materials that can be extracted by contact with synthetic urine (so-called extractables). An example of such SAP particles is starch graft polyacrylate hydrogel, available as IM1000® from Hoechst-Celanese of Portsmouth, VA. Other examples of hydrogels containing SAP particles include, but are not limited to, those sold under the trademarks SANWET™, available from Sanyo Kasei Kogyo Kabushiki of Japan; SUMIKA GEL™ available from Sumitomo Kagaku Kabushiki Haishi of Japan; and FAVOR™ available from Stockhausen of Garyville, LA; and ASAP™ available from BASF of Aberdeen, MS.

According to one preferred embodiment, the absorbent core contains (a) SAP particles and (b) fluff fibers, matrix fibers, the fibers of the present invention, or any combination of the foregoing. The fibers provide structural integrity and avenues for the passage of fluid through the absorbent core.

According to another embodiment, the absorbent core contains from about 30 to about 70% by weight of SAP particles and from about 70 to about 30% by weight of the cellulose fibers of the present invention, based on 100% total weight of the absorbent core.

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Generally, the weight density of the absorbent core ranges from about 0.15 to about 0.25 g/cm³.

According to yet another embodiment, the absorbent core comprises SAP particles and reversible crosslinked fibers of the present invention. According to one preferred embodiment, the reversible crosslinked fibers are crosslinked with oxalic acid, sodium chloroacetate, or a mixture thereof. Generally, the absorbent core contains from about 30 to about 70% by weight of SAP particles and from about 70 to about 30% by weight of reversible crosslinked fibers of the present invention, based on 100% total weight of the absorbent core. According to another preferred embodiment, the acquisition and/or distribution layers and the absorbent core contain fibers crosslinked with oxalic acid.

The absorbent structure of the present invention may be incorporated into disposable and non-disposable absorbent articles, such as diapers, adult incontinence pads and feminine hygiene articles. The absorbent article can include a liquid pervious top-sheet above the acquisition and/or distribution layer, whose function is to allow the passage of fluid to the acquisition and/or distribution layer, and a liquid impervious back-sheet, whose function is to contain the fluid and to prevent it from passing through the absorbent article to the garment of the wearer of the absorbent article.

According to one embodiment, the absorbent structure of the present invention is incorporated into a disposable infant diaper which generally includes a front waistband area, a rear waistband area, and a crotch region there between. The structure of the diaper generally includes a liquid pervious top-sheet, a liquid impervious back-sheet, the absorbent structure, elastic members, and securing tabs. Representative disposable diaper designs may be found, for example, in U.S. Patent Nos. 4,935,022 and 5,149,335.

According to another embodiment, the absorbent structure of the present invention is incorporated into a feminine hygiene pad, such as that disclosed in U.S. Patent No. 5,961,505.

The following examples illustrate the invention without limitation. All parts and percentages are given by weight unless otherwise indicated. All the crosslinking agents, crosslinking facilitators, and other chemical reagents used in the examples are available from Aldrich Chemical Company of Milwaukee, WI.

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<u>Measurement of Capillary and Desorption Pressures and Saturated Capacity</u>

The capillary absorption and desorption pressures were determined according to the procedure described in "Capillary Sorption Equilibria in Fiber Masses", A. A. Burgeni and C. Kapur, *Textile Research Journal*, 37:356-366 (1967). This procedure is described in detail below.

A 0.75 g sample of individualized fibers is formed into a disc approximately 55-60 mm in diameter. The sample is placed on the frit of a 150 ml coarse frit Pyrex® glass funnel, Corning No. 36060 (available from VWR of Suwanee, GA). A weight sufficient to apply 0.22 psi pressure, of diameter comparable to that of the sample is placed on the sample. The bottom of the funnel is attached to an adapter with decreasing diameter such that Tygon® tubing No. R – 3603, approximately two feet in length, is attached to the adapter on one end, and on the other end to a fluid reservoir resting on an electronic scale capable of measuring 0.01 g. The tubing is attached to the side of the reservoir at the bottom. The fluid reservoir contains 0.9% saline solution. The height of the saline solution in the fluid reservoir is approximately 1 inch above the tubing attachment. The tubing is filled with saline, as is the funnel below the frit, such that the frit is damp with the saline, but no saline is above the frit. The saline column from the reservoir to the frit is continuous without any air in the column.

The absorption cycle is as follows. Starting at a height of, for example, 20, 30, or 80 cm above the level of saline in the reservoir, the sample is allowed to absorb saline to equilibrium or steady state. Steady state is determined as no change in the weight of saline on the electronic scale beneath the reservoir greater than 0.04 g over a period of one minute. When steady state is achieved, the sample is lowered 5 cm closer to the level of saline in the reservoir and held there until equilibrium is achieved. The sample is lowered another 5 cm and the procedure repeated. When the sample is at equilibrium at the same height as the saline level in the reservoir, the sample is subjected to a desorption cycle by reversing the procedure, *i.e.*, by moving the sample upward in 5 cm increments.

The weight of saline in the sample at the same level as the saline in the reservoir is the saturated capacity of the sample. The height (reported in cm) of the sample above the saline level in the reservoir at 50% of saturated capacity on the downward (absorption) cycle (median absorption pressure) and on the upward (desorption) curve (median desorption pressure) are determined by interpolation. The value of saturated capacity is reported in table as grams of saline per gram of sample.

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<u>Examples 1-8</u>

Examples 1-8 in Table 1 were prepared as follows.

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley
Fluff™ fibers from Buckeye Technologies Inc. of Memphis, TN, were slurried in water and
refined in a valley beater at ambient temperature and pressure to the appropriate degree of
freeness. The fibers were centrifuged, separated by hand, and air dried to 60% solids. The
fibers were crosslinked with the appropriate concentration of citric acid (dry fiber basis) by
spraying the fibers with an aqueous solution of citric acid having sufficient dilution to bring
the sheet to 40 % solids content. The fibers were then air dried to 60% solids content,
fluffed, and dried to constant weight and heated for an additional 30 minutes at the
temperature indicated in Table 1.

The water retention value, saturation capacity, capillary absorption pressure,
and capillary desorption pressure of the cured cellulose fibers were determined. The water
retention value of the cured cellulose fibers was determined according to the procedure
described in TAPPI Useful Methods, UM 256. The capillary absorption and desorption
pressures were determined according to the procedure described above.

The results are shown in Table 1.

<u>Comparative Example 9</u>

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley
Fluff™ fibers from Buckeye Technologies Inc. of Memphis, TN, were dried at 100° C. The
water retention value, saturation capacity, capillary absorption pressure, and capillary
desorption pressure of the cured cellulose fibers were determined as described in Example 1.

The results are shown in Table 1 below.

<u>Comparative Example 10</u>

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley
Fluff™ fibers from Buckeye Technologies Inc., were sprayed with sufficient water to
produce a 40% solids content. The fibers were air dried to 60% solids content, mechanically
fluffed, and dried to constant weight at 150° C. The fibers were then heated for an additional
30 minutes at the same temperature. The water retention value, saturation capacity, capillary

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absorption pressure, and capillary desorption pressure of the cured cellulose fibers were determined as described in Example 1.

The results are shown in Table 1.

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Comparative Example 11

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff[TM] fibers from Buckeye Technologies Inc., were slurried in water and refined in a valley beater to a freeness of approximately 500 ml CSF. The fibers were centrifuged, separated by hand, air dried to 60% solids content, fluffed, and dried to constant weight at 150° C. The

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fibers were then heated for an additional 30 minutes at 150° C. The water retention value, saturation capacity, capillary absorption pressure, and capillary desorption pressure of the cured cellulose fibers were determined as described in Example 1.

The results are shown in Table 1.

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Comparative Example 12

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff[TM] fibers from Buckeye Technologies Inc., were slurried in water and refined in a valley beater to a freeness of approximately 500 ml CSF. The fibers were centrifuged, separated by hand, and air dried to 60% solids. The cellulose fibers were sprayed with an aqueous sulfuric

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acid solution at pH 3 to 40% solids content in order to adjust the pH of the fiber water mixture to the same pH as that observed in Example 1, when the fibers were treated with aqueous citric acid. The fibers were air dried to 60% solids content, fluffed, and dried to constant weight at 150° C. The fibers were then heated for an additional 30 minutes at 150° C. The water retention value, saturation capacity, capillary absorption pressure, and capillary

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desorption pressure of the cured cellulose fibers were determined as described in Example 1.

The results are shown in Table 1.

Comparative Example 13

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley

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Fluff[TM] fibers from Buckeye Technologies Inc., were crosslinked with 5% citric acid (dry fiber basis) by spraying the fibers with an aqueous solution of citric acid having sufficient dilution to bring the sheet to 40 % solids content. The sheet was air dried to 60% solids content, mechanically fluffed, dried to constant weight at 150° C. The sheet was then heated

for an additional 30 minutes at 150° C. The water retention value, saturation capacity, capillary absorption pressure, and capillary desorption pressure of the cured cellulose fibers were determined as described in Example 1.

The results are shown in Table 1.

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Comparative Example 14

The procedure described in Comparative Example 13 was repeated, except the fibers were crosslinked with 10% citric acid instead of 5% citric acid.

The results are shown in Table 1.

Table 1

Example	Freeness (ml CSF)	Crosslinked (% Citric Acid) (Dry Fiber Basis)	Cure Temperature (°C)	WRV (%)	Saturation Capacity (g/g)	Absorption Pressure at 50% of Saturated Capacity (cm)	Desorption Pressure at 50% of Saturated Capacity (cm)
Example 1	500	5	150	44.8	8.2	3.9	13.4
Example 2	500	10	150	38.1	8.7	4.4	13.5
Example 3	300	5	150	42.7	8.2	5.3	14.7
Example 4	300	10	150	41.5	8.6	5.1	14.9
Example 5	500	5	175	36.7	8.5	4.4	14.0
0Example 6	500	10	175	29.5	8.3	3.1	11.9
Example 7	300	5	175	33.4	7.9	4.2	15.0
Example 8	300	10	175	29.9	7.5	3.0	13.3
Comparative Example 9	740	-	100	83.0	9.3	12.3	30.5
Comparative Example 10	740	-	150	73.6	11.0	12.0	27.4
Comparative Example 11	500	-	150	92.1	8.2	8.1	20.7
Comparative Example 12	500	-	150	78.6	8.3	5.3	16.3
Comparative Example 13	740	5	150	43.3	12.3	-	17.9
Comparative Example 14	740	10	150	38.4	12.1	6.7	18.4

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As shown by the results in Table 1, the refined and crosslinked fibers exhibited lower WRVs and desorption pressures than similar unrefined and crosslinked fibers.

<center>Example 15</center>

Refining

An aqueous slurry containing 2.75-3.25% by weight of never dried Foley Fluff™ fibers, available from Buckeye Technologies Inc., was prepared. The aqueous slurry was passed through a Bauer Model No. 444, 24" pump through refiner, at ambient temperature and pressure. The Bauer refiner plates were No. A24313.

The refiner was operated at a current of 178 amps and a slurry flow rate of 255 gallons per minute. These conditions produced loading of 30-60 horsepower hours per bone dry short ton of fibers. The fibers produced had a freeness of 680 ml CSF.

Demineralizing

The refined pulp slurry was pumped to a false bottom tank at a consistency of 2.75-3.25%. While stirring the pulp slurry, sulfuric acid was added until a nominal pH of 2.0 was obtained. After at least 10 minutes of stirring, the aqueous slurry was allowed to de-water through the false bottom screen for a minimum of 3 hours. The slurry was then diluted with sodium softened water to 2.0% consistency and the pH was adjusted to 4.5-5.0.

Sheeting

Pulp sheeting was performed on a paper machine available from Sandy Hill Corporation of Hudson Falls, NY. The deckle (sheet width) was a maximum of 36 inches. The 2% pulp slurry was pumped through a stuff box and a basis weight valve and into a white water silo at a controlled flow rate. The temperature in the silo was increased to 130-150° F with direct steam and the slurry in the silo was diluted with white water to a consistency of 1.0-1.25%.

The slurry was then fed into a paper machine headbox and moved onto a moving wire at the Fordrinier section of the paper machine. Natural drainage and vacuum assisted drainage were provided until the formed sheet exited the couch press at about 32% consistency. After formation of the sheet, but before the couch press, the wet sheet was

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trimmed with two jets of water to deckle of 24 inches. The sheet of fibers having a consistency of about 32% then passed through two wet presses where further water removal and sheet densification occurred. After exiting the second wet press, the sheet of fibers entered a first dryer section at approximately 48% consistency. In the first dryer section, the

5 pulp sheet passed over thirteen rotating steam cans at approximately 300-325° F. The pulp sheet was then passed over eight rotating steam cans in a second dryer section and exited the dryer at a moisture content of 4-8%. The sheet was wound into a roll at a deckle of about 23 inches. The basis weight of this rolled sheet was approximately 0.126 pound per square foot and the density was approximately 0.60 grams per cubic centimeter.

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Slitting

The rolls of pulp were rewound onto a new core and slit into smaller rolls, each 10 inches wide.

15 Chemical Application

A 10 inch wide roll of the pulp sheet was unwound and slowly passed through a puddle press. At the nip of the puddle press was an aqueous solution of citric acid and sodium hypophosphite. The sodium hypophosphite moderates pulp darkening at high temperature. The weight concentrations of citric acid and sodium hypophosphite in the

20 flooded nip were approximately 14% and 7%, respectively. Through the puddle press, the sheet absorbed enough of the aqueous solution to reach a moisture content of about 40%.

Sheet Disintegration and Fluffing

Following the puddle press, the sheet was picked apart into smaller pieces

25 through a shredder, a pre-breaker and a picker. The disintegrated pulp was then blown into the inlet of a Sunds Defibrator Model 3784 RO Fluffer, available from Sunds Defibrator, AB of Sundsvall, Sweden, with a gap setting of 5.5 mm. The defibrator fluffed the pulp into masses of separated fibers. The fluffed pulp was swept out of the RO Fluffer with a high velocity stream of hot air at approximately 380° F.

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Drying and Curing

The hot air flow that conveyed the fluffed fiber out of the RO Fluffer was boosted with a fan through a flash dryer where all or almost all of the water in the fibers was evaporated. The dried pulp fell onto a mechanical inlet conveyor forming a low density high bulk bed on the conveyor. The fibers were then transported into a Proctor & Schwartz K16476 tunnel dryer, available from Proctor & Schwartz, Inc. of Horsham, PA. Through a series of hot circulating air flows, the fluffed fiber bed was heated and then allowed to cool through three chambers in the dryer. In chamber 1, the bed temperature reached 325-330° F. In chamber 2, the bed temperature increased to 385-390° F. In chamber 3, the bed temperature decreased to 355-360° F. The total time in the tunnel dryer was approximately 11.5 minutes.

Baling

The crosslinked fibers fell off the conveyer from the exit side of the tunnel dryer into a baler model no. 3445, available from American Baler Company of Bellevue, OH, where the material was compressed into bales weighing approximately 70-80 pounds.

Example 16

The samples in Table 2 were prepared as follows.

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff™ fibers from Buckeye Technologies Inc. of Memphis, TN, were slurried in water and refined in a Bauer Model No. 444, 24" pump through refiner, at ambient temperature and pressure to the appropriate degree of freeness. Optionally, the fibers were washed with dilute sulfuric acid (acid wash) to remove minerals. The refined fibers were sheeted and dried.

A piece of the sheet was submerged in a tray containing a solution of the crosslinking agent and oxalic acid. The piece was then flipped over and submerged in a second solution of the crosslinking agent and oxalic acid. The combined solutions contained 10% of the crosslinking agent (dry fiber basis) and 5% oxalic acid (dry fiber basis). The solutions had sufficient dilution to bring the piece to 40 % solids content. The piece was placed in a sealed polyethylene bag for 1 hour. The fibers were air dried to 60% solids content, fluffed, and dried to constant weight and heated for an additional 30 minutes at 175° C.

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The water retention value, saturation capacity, capillary absorption pressure, and capillary desorption pressure of the cured cellulose fibers were determined. The water retention value of the cured cellulose fibers was determined according to the procedure described in TAPPI Useful Methods, UM 256. The capillary absorption and desorption pressures were determined according to the procedure described above.

The results are shown in Table 2.

Table 2

Sample	Crosslinking Agent	Freeness (ml CSF)	Acid Washed	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity	Carboxyl (meq/kgm)
a	Succinic Acid	740	No	31.5	13.9	6.8	14.7	343.1
b	Succinic Acid	570	No	36.3	13.5	7.2	17.3	294.1
c	Succinic Acid	570	Yes	36.0	13.7	7.2	16.1	303.9
d	Adipic Acid	740	No	36.0	13.5	7.4	16.4	228.6
e	Adipic Acid	570	No	39.1	12.6	7.0	16.4	190.6
f	Adipic Acid	570	Yes	40.0	13.1	7.8	18.3	179.3
g	Pimelic Acid	740	No	35.4	14.7	6.4	17.5	231.4
h	Pimelic Acid	570	No	39.4	13.9	6.9	17.7	209.0
i	Pimelic Acid	570	Yes	37.9	12.2	7.3	17.3	200.0
j	Malonic Acid	740	No	32.8	16.1	7.2	18.9	75.6
k	Malonic Acid	570	No	36.5	11.6	6.1	16.8	78.2
l	Malonic Acid	570	Yes	37.7	12.7	7.1	15.8	85.5

Example 17

The samples in Table 3 were prepared as follows.

Unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff[TM] fibers from Buckeye Technologies Inc. of Memphis, TN, were slurried in water and refined in a Bauer Model No. 444, 24" pump through refiner, at ambient temperature and pressure to the appropriate degree of freeness. The refined fibers were sheeted and dried. If the fibers were wet lap, they were then centrifuged.

A piece of the sheet was submerged in a tray containing a solution of the crosslinking agent, sodium hypophosphite, and, optionally, oxalic acid. The piece was then flipped over and submerged in a second solution of the crosslinking agent, sodium hypophosphite, and, optionally, oxalic acid. The combined solutions contained 10% of the crosslinking agent (dry fiber basis), 5% sodium hypophosphite (dry fiber basis) and, optionally, 1% oxalic acid (dry fiber basis). The solutions had sufficient dilution to bring the piece to 40 % solids content. The piece was placed in a sealed polyethylene bag for 1 hour. The fibers were air dried to 60% solids content, fluffed, and dried to constant weight and heated for an additional 30 minutes at 175° C.

The water retention value, saturation capacity, capillary absorption pressure, and capillary desorption pressure of the cured cellulose fibers were determined. The water retention value of the cured cellulose fibers was determined according to the procedure described in TAPPI Useful Methods, UM 256. The capillary absorption and desorption pressures were determined according to the procedure described above.

The results are shown in Table 3.

Table 3

Sample	Crosslinking Agent	Concentration of Oxalic Acid (% w/w)	Freeness (ml CSF)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity	Carboxyl (meq/kgm)
a	Oxalic Acid	-	740	48.3	12.0	6.2	18.9	292.5
b	Maleic Acid	-	740	33.8	15.3	4.7	16.4	529.5
c	Succinic Acid	-	740	38.2	14.0	5.3	16.7	414.8
d	Adipic Acid	-	740	45.7	13.4	5.7	17.2	343.9
e	Succinic Acid	1	740	37.8	14.5	4.6	14.9	432.2
f	Adipic Acid	1	740	45.3	13.0	6.1	17.8	356.3
g	Citric Acid	-	740	33.1	15.6	4.4	15.2	442.0
h	Oxalic Acid	-	680	47.3	10.5	6.5	18.4	287.7
i	Maleic Acid	-	680	34.2	14.4	4.2	15.0	530.2
j	Succinic Acid	-	680	38.0	12.8	5.8	16.6	436.8
k	Adipic Acid	-	680	49.6	11.8	5.4	15.6	351.3
l	Succinic Acid	1	680	37.0	14.0	4.4	14.3	424.6
m	Adipic Acid	1	680	45.8	12.3	6.3	17.7	358.5
n	Citric Acid	-	680	31.3	15.8	3.7	13.5	450.3

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Sample	Crosslinking Agent	Concentration of Oxalic Acid (% w/w)	Freeness (ml CSF)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity	Carboxyl (meq/kgm)
o	Oxalic Acid	-	570 (Wet Lap)	59.9	3.2	5.2	12.7	432.0
p	Maleic Acid	-	570 (Wet Lap)	41.6	5.1	3.8	7.7	474.0
q	Succinic Acid	-	570 (Wet Lap)	34.8	8.8	3.2	8.3	466.1
r	Adipic Acid	-	570 (Wet Lap)	50.1	9.0	3.4	8.7	487.5
s	Succinic Acid	1	570 (Wet Lap)	36.1	8.8	3.3	8.7	415.4
t	Adipic Acid	1	570 (Wet Lap)	44.2	8.6	3.3	8.9	422.1
u	Citric Acid	-	570 (Wet Lap)	35.0	9.7	3.3	9.2	468.6
v	Oxalic Acid	-	570	49.6	11.7	5.3	15.8	405.8
w	Maleic Acid	-	570	41.5	12.4	5.9	16.7	450.7

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Sample	Crosslinking Agent	Concentration of Oxalic Acid (% w/w)	Freeness (ml CSF)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity	Carboxyl (meq/kgm)
x	Succinic Acid	-	570	37.2	13.4	4.8	15.1	510.1
y	Adipic Acid	-	570	44.9	11.3	5.3	16.0	328.0
z	Succinic Acid	1	570	40.7	12.0	5.4	16.2	455.5
aa	Adipic Acid	1	570	48.6	11.4	5.9	16.7	371.0
bb	Citric Acid	-	570	39.9	13.7	3.8	12.9	481.1

Example 18

The samples described in Table 4 below were prepared as follows.

Stock solutions of the crosslinking agents and crosslinking facilitators identified in Table 4 were prepared by dissolving the indicated amounts of crosslinking agent and crosslinking facilitator in 22.5 g of distilled water.

Samples were treated with the appropriate solution as follows. A 15 g (dry basis) sample of sheeted, unrefined cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff™ fibers from Buckeye Technologies Inc. of Memphis, TN, was treated with the appropriate stock solution containing the crosslinking agent and crosslinking facilitator. This reduced the fiber solids content of the mixture to 40%. The mixture was placed in a sealed container for 60 minutes at ambient temperature and then air dried to 60% solids content. The fibers were mechanically separated, individualized, and fluffed in a laboratory fluffer and dried to constant weight in a forced air oven at 175° C. The fibers were cured for 30 minutes at the same temperature.

A control was prepared as follows. A dry unrefined sheet of cellulose fibers having a freeness of 740 ml CSF, available as Foley Fluff™ fibers from Buckeye Technologies Inc. of Memphis, TN, was diluted to a 40% solids content with water. The pH of the mixture was adjusted to 3 with sulfuric acid. The stock solutions applied to the fibers in the procedure above typically have a pH of 3. The mixture was then placed in a sealed container for 60 minutes at ambient temperature and then air dried to a 60% solids content. The fibers were mechanically separated, individualized and fluffed in a laboratory fluffer and dried to constant weight in a forced air oven at 175 ° C. The fibers were heated for an additional 30 minutes at the same temperature.

Fibers from an acquisition-distribution layer of a Pampers® disposable diaper, available from Proctor and Gamble of Cincinnati, OH, were obtained and used as a second control.

The water retention value, saturation capacity, capillary absorption pressure, and capillary desorption pressure of the fibers were determined. The water retention value of the cellulose fibers was determined according to the procedure described in TAPPI Useful Methods, UM 256. The capillary absorption and desorption pressures and saturated capacity were determined by the procedure described above.

The results are shown in Table 4.

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Table 4

Sample	Crosslinking Agent	Amount of Crosslinking Agent in Stock Solution (g)	Crosslinking Facilitator	Amount of Crosslinking Facilitator in Stock Solution (g)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity
a	None (Control)	-	None	-	59.4	13.6	10.7	24.6
b	Pampers® (Control)	-	-	-	44.9	6.7	4.4	18.2
c	Sodium Chloroacetate	0.75	None	-	49.8	12.7	9.5	22.2
d	Sodium Chloroacetate	1.5	None	-	44.6	11.1	8.2	18.5
e	Oxalic Acid	0.15	None	-	47.2	12.9	9.1	23.6
f	Oxalic Acid	0.75	None	-	42.9	13.6	8.6	21.5
g	Oxalic Acid	1.5	None	-	38.5	15.3	7.6	17.8
h	Succinic Acid	1.5	None	-	39.8	14.2	7.5	20.4
i	Succinic Acid	1.5	Oxalic Acid	0.15	33.8	14.3	7.7	16.6
j	Succinic Acid	1.5	Oxalic Acid	0.75	31.5	13.9	6.8	14.7
k	Adipic Acid	1.5	None	-	50.5	14.1	8.0	22.3
l	Adipic Acid	1.5	Oxalic Acid	0.15	38.9	13.7	8.6	19.8
m	Adipic Acid	1.5	Oxalic Acid	0.75	36.0	13.5	7.4	16.4
n	Malonic Acid	1.5	None	-	39.5	13.5	7.8	22.8
o	Malonic Acid	1.5	Oxalic Acid	0.75	32.8	16.1	7.2	18.9

Sample	Crosslinking Agent	Amount of Crosslinking Agent in Stock Solution (g)	Crosslinking Facilitator	Amount of Crosslinking Facilitator in Stock Solution (g)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50% of Saturated Capacity	Desorption Pressure (cm) @ 50% of Saturated Capacity
p	Glutaric Acid	1.5	None	-	37.1	15.2	7.5	23.4
q	Glutaric Acid	1.5	Oxalic Acid	0.75	33.0	15.9	7.3	21.9
r	Pimelic Acid	1.5	None	-	42.7	14.8	7.4	23.1
s	Pimelic Acid	1.5	Oxalic Acid	0.75	35.4	14.7	6.4	17.5
t	Suberic Acid	1.5	None	-	56.0	13.7	8.3	25.7
u	Suberic Acid	1.5	Oxalic Acid	0.75	41.0	14.2	7.2	21.4
v	Phthalic Acid	1.5	None	-	55.4	13.9	8.4	26.3
w	Phthalic Acid	1.5	Oxalic Acid	0.75	42.5	13.0	9.0	24.6
x	Tetrahydro-phthalic Acid	1.5	None	-	53.4	12.6	8.4	22.5
y	Tetrahydro-phthalic Acid	1.5	Oxalic Acid	0.75	39.4	13.3	7.4	19.9
z	Fumaric Acid	1.5	None	-	44.5	13.9	9.1	24.1
aa	Fumaric Acid	1.5	Oxalic Acid	0.75	41.5	12.7	8.4	19.2
bb	Glycolic Acid	1.5	None	-	47.8	13.3	8.7	22.0
cc	Glycolic Acid	1.5	Oxalic Acid	0.75	39.6	14.0	7.3	17.4
dd	Tartaric Acid	1.5	None	-	34.3	13.7	7.3	18.5
ee	Tartaric Acid	1.5	Oxalic Acid	0.75	32.2	13.1	7.3	18.3
ff	Malic Acid	1.5	None	-	31.6	14.5	6.9	19.0
gg	Malic Acid	1.5	Oxalic Acid	0.75	30.1	14.0	6.6	18.7

Sample	Crosslinking Agent	Amount of Crosslinking Agent in Stock Solution (g)	Crosslinking Facilitator	Amount of Crosslinking Facilitator in Stock Solution (g)	WRV (%)	Saturated Capacity (g/g)	Absorption Pressure (cm) @ 50 % of Saturated Capacity	Desorption Pressure (cm) @ 50 % of Saturated Capacity
hh	Saccharic Acid	1.5	None	-	49.6	13.4	9.4	22.8
ii	Saccharic Acid	1.5	Oxalic Acid	0.75	41.1	11.4	8.2	19.4
jj	Mucic Acid	1.5	None	-	55.9	12.4	9.9	19.0
kk	Mucic Acid	1.5	Oxalic Acid	0.75	40.3	11.9	8.4	17.1
ll	Aspartic Acid	1.5	None	-	55.2	13.9	9.5	26.6
mm	Aspartic Acid	1.5	Oxalic Acid	0.75	37.5	14.6	6.5	16.5
nn	Glutamic Acid	1.5	None	-	52.8	13.8	7.8	25.1
oo	Glutamic Acid	1.5	Oxalic Acid	0.75	37.4	14.2	7.0	17.3
pp	EDTA	1.5	None	-	50.7	12.1	8.6	21.9
qq	EDTA	1.5	Oxalic Acid	0.75	39.7	12.1	8.0	18.4

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Example 19

A sample of crosslinked fibers was prepared by the method described in Example 18 with 1.5 g sodium chloroacetate. A second sample was prepared with 1.5 g oxalic acid instead of sodium chloroacetate.

For comparison purposes, a sample of crosslinked fibers was prepared by the procedure in Example 18 using a stock solution containing 10% by weight of citric acid.

The samples were subjected to a first capillary absorption-desorption cycle by the procedure described above. The samples were then subjected to a second capillary absorption-desorption cycle by the same procedure. The observed absorption and desorption pressures for both cycles are shown in Table 5 below.

This test was repeated with uncrosslinked Foley Fluff™ fibers.

Table 5

	Crosslinking Agent			
	None	Oxalic Acid	Sodium Chloroacetate	Citric Acid
First Cycle				
Saturated Capacity (g/g)	13.6	15.3	12.2	18.5
Absorption Pressure (cm) @ 50% of Saturated Capacity	10.7	7.6	10.5	7.0
Desorption Pressure (cm) @ 50% of Saturated Capacity	24.6	17.8	18.2	13.5
Second Cycle				
Saturated Capacity (g/g)	11.8	13.1	9.6	16.5
Absorption Pressure (cm) @ 50% of Saturated Capacity	> 30	> 30	> 30	15.3
Desorption Pressure (cm) @ 50% of Saturated Capacity	25.8	29.3	24.7	18.4

Example 20

Samples as described in Example 19 were prepared and subjected to two capillary absorption-desorption cycles. The samples were dried overnight in a forced air

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oven at 105° C. Alternatively, the samples were dried to constant weight at 105° C and
heated for an additional 30 minutes (cured) at 175° C. The saturated capacity and absorption
and desorption pressures of the samples was determined.

The results are shown in Table 6.

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Table 6

	Crosslinking Agent			
	None	Oxalic Acid	Sodium Chloroacetate	Citric Acid
Dried at 105° C				
Saturated Capacity (g/g)	12.8	12.3	10.3	17.1
Absorption Pressure (cm) @ 50% of Saturated Capacity	12.2	8.6	11.5	7.1
Desorption Pressure (cm) @ 50% of Saturated Capacity	24.7	18.1	19.8	14.3
Cured at 175° C				
Saturated Capacity (g/g)	11.3	11.2	9.5	14.4
Absorption Pressure (cm) @ 50% of Saturated Capacity	13.0	6.8	12.6	7.2
Desorption Pressure (cm) @ 50% of Saturated Capacity	24.6	18.1	19.6	13.7

Example 21

10   Chemical Application

An aqueous solution of oxalic acid and sodium hypophosphite was prepared
by mixing 151 pounds of a 10% by weight oxalic acid solution, 15 pounds of a 50% by
weight sodium hypophosphite solution, and 1 pound of water.

A 10 inch wide roll of Foley Fluff[TM] fibers, available from Buckeye
15   Technologies Inc., was unwound and slowly passed through a puddle press. Flooded at the
nip of the puddle press was the aqueous solution of oxalic acid and sodium hypophosphite.
The sodium hypophosphite moderates pulp darkening at high temperature. Through the
puddle press, the sheet absorbed enough of the aqueous solution to reach a moisture content

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of about 47%, based upon 100% total weight of dry fibers. The treated sheet also contained about 10% by weight of oxalic acid and 5% sodium hypophosphite, based upon 100% total weight of dry fibers.

5   Sheet Disintegration and Fluffing

Following the puddle press, the sheet was picked apart into smaller pieces through a shredder, a pre-breaker and a picker. The disintegrated pulp was then blown into the inlet of a Sunds Defibrator Model 3784 RO Fluffer, available from Sunds Defibrator, AB of Sundsvall, Sweden, with a gap setting of 5.5 mm. The defibrator fluffed the pulp into 10   masses of separated fibers. The fluffed pulp was swept out of the RO Fluffer with a high velocity stream of hot air at approximately 380° F.

Drying and Curing

The hot air flow that conveyed the fluffed fiber out of the RO Fluffer was 15   boosted with a fan through a flash dryer where all of the water in the fibers was evaporated. The dried pulp fell onto a mechanical inlet conveyor forming a low density high bulk "bed" on the conveyor. The fibers were then transported into a Proctor & Schwartz K16476 tunnel dryer, available from Proctor & Schwartz, Inc. of Horsham, PA. Through a series of hot circulating air flows, the fluffed fiber bed was heated through three chambers in the dryer. In 20   chamber 1, the bed temperature reached 330-340° F. In chamber 2, the bed temperature increased to 375-385° F. In chamber 3, the bed temperature decreased to 355-360° F. After the three heating zones, the fiber bed passed through one last insulated chamber with no additional heat being added. The total time in the tunnel dryer was approximately 11.5 minutes.

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Baling

The crosslinked fibers fell off the conveyer from the exit side of the tunnel dryer into a baler model no. 3445, available from American Baler Company of Bellevue, OH, where the material was compressed into bales weighing approximately 85-100 pounds.

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All references cited herein are incorporated by reference. To the extent that a conflict may exist between the specification and the reference the language of the disclosure made herein controls.